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औद्योगिक उद्देश्यों के लिए सिंथेटिक  
डिटर्जेंट — विशिष्टि

( तीसरा पुनरीक्षण )

**Synthetic Detergents for Industrial  
Purposes — Specification**

( *Third Revision* )

ICS 71.100.40

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## FOREWORD

This Indian Standard (Third Revision) was adopted by Bureau of Indian Standards, after the draft finalized by the Soaps and Other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Synthetic detergents or non-soapy detergents (NSD), as they are usually termed, are products specially formulated to promote the development of detergency, comprise essential components (like surface active agents), and generally, complementary components like builders, etc. The NSD produced in this country are mainly of the alkyl aryl type, such as sodium salt of dodecylbenzene sulphonic acid and thus they are different from soaps, the other class of surface active agents, which are mainly sodium salts of higher fatty acids. The limitations of soaps for use in hard water areas have led to the development of synthetic detergents.

This standard was first published in 1968 and subsequently revised in 1977 and 2002. During its revision in 1977, the requirement for matter insoluble in alcohol and non-detergent organic matter were dropped, the limit for moisture content was reduced and the requirement for active ingredient for Type 2 of the material was increased from 30 to 40 percent and that for Type 1 was reduced from 40 to 35 percent in view of prevailing industrial practice. The method for determination of active matter by cationic titration was also incorporated.

During second revision in 2002, the declaration of ingredients used in the manufacture of detergents had been incorporated in marking clause.

Presence of phosphorous in excess in water body is known to cause eutrophication leading to algal growth, thereby posing risks to aquatic life. Apart from fertilizer industry, phosphorous based compounds (for example, phosphates, phosphonates etc.) used in detergent products can contribute to the above. Therefore, in pursuit of minimizing the environmental impact, the committee responsible for formulation of this standard has decided to revise it again. Hence, beside amalgamation of amendment (after review), the maximum limit of phosphate has been restricted in this revision. Performance requirements like detergency and ash built up have also been incorporated in this revision. Further, the use of Zeolite, which is a non-phosphate based builder and use of co-builders enzymes (bio-based actives) for detergent have been recommended as an alternative to phosphate based builder. Tri-sodium citrate has also been incorporated in list of conventional builders and additives since it has the potential to replace phosphates and is more environment friendly than the phosphates.

It is necessary that the raw materials used in the formulation of detergents are such that in the concentration in which they will be present in the finished product, after interaction between them are free from any harmful effects. For determining the suitability of a new formulation or of a new raw material used in formulations for skin safety, necessary tests as prescribed in IS 11601 : 2002 Methods of safety evaluation of synthetic detergents — Tests for skin irritation and sensitization potential of synthetic detergents need to be followed.

A scheme for labelling environment friendly products to be known as ECO Mark has been introduced at the instance of Ministry of environment and Forest and Climate Change (MoEF & CC). The ECO Mark shall be administered by the Bureau of Indian Standards (BIS) under the *BIS Act*, 2016 as per the Resolution No 71 dated 20 February 1991 published in the Gazette of the Government of India For a product to be eligible for ECO Mark, it shall carry the standard mark of BIS for quality besides meeting additional optional environmentfriendly (EF) requirements.

This standard covers **6.1** which calls for an agreement between the purchaser and the supplier.

The composition of the technical committee responsible for formulation of this standard is given in Annex J.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## *Indian Standard*

# SYNTHETIC DETERGENTS FOR INDUSTRIAL PURPOSES — SPECIFICATION

( *Third Revision* )

### 1 SCOPE

This standard prescribes requirements, methods of sampling and test for synthetic anionic detergents for industrial purposes.

### 2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
286 : 2018	Methods of sampling and test for soaps ( <i>third revision</i> )
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
4262 : 2002	Sulphuric acid — Code of safety ( <i>first revision</i> )
4707 (Part 1) : 2020	Classification for cosmetics raw materials and adjuncts: Part 1 Colourants ( <i>fourth revision</i> )
4707 (Part 2) : 2017	Classification for cosmetic raw materials and adjuncts: Part 2 List of raw materials generally not recognized as safe for use in cosmetics ( <i>fourth revision</i> )
4905 : 2015	Random sampling and randomization procedures ( <i>first revision</i> )
4955 : 2020	Household laundry detergent powders — Specification ( <i>fifth revision</i> )
5785 (Part 4) : 1976	Methods for performance tests for surface active agents: Part 4 relative detergency ( <i>first revision</i> )
7597 : 2001	Surface active agents — Glossary of terms ( <i>first revision</i> )

### *IS No.*

### *Title*

8401 : 1994	Alkyl benzene sulphonic acid (acid slurry) — specification ( <i>first revision</i> )
11601 : 2002	Methods of safety evaluation of synthetic detergents — Tests for skin irritation and sensitization potential of synthetic detergents ( <i>first revision</i> )
13933 : 1995	Method of test for ready biodegradability of surface active agents (modified sturm test)
15267 : 2003	Zeolite detergentgrade — Specification

### 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7597 shall apply.

### 4 TYPES

The material shall be of following three types based on its physical form, namely:

- a) *Type 1* — Powder or flakes;
- b) *Type 2* — Paste; and
- c) *Type 3* — Liquid.

### 5 REQUIREMENTS

#### 5.1 Description

The active ingredient shall be the sodium salt of alkyl benzene sulphonic acid conforming to IS 8401. The formulation may contain one or more of the builders or additives given in Annex A or any other builders or additives keeping in view the end-use of the product.

**5.2** The material shall be in the form of free flowing powders, paste or clear liquid, free from visible dirt and impurities. It shall not give any unpleasant odour. It shall have good cleaning and lathering properties. The material shall not contain any microbeads or other synthetic abrasive material.

**5.3** The material may contain permitted colour as given in IS 4707 (Part 1). The material shall not contain any ingredient above the limit as given in IS 4707 (Part 2). The material shall pass the test for skin irritant and sensitization potential when evaluated as per the method prescribed in IS 11601.

**5.4** The material shall also comply with the requirements given in Table 1.

## 5.5 Additional Requirements for ECO Mark

### 5.5.1 General Requirements

**5.5.1.1** The product shall conform to the requirements for quality, safety and performance prescribed under **5.1** to **5.4** for different types except that for phosphate content which shall be substituted with alternate environment friendly builder(s) to maintain similar detergency when tested according to the method prescribed in IS 5785 (Part 4) (*see also 5.5.2.1*).

**5.5.1.2** The manufacturer shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of *Water (Prevention and Control of Pollution) Act, 1974* and *Air (Prevention and Control of Pollution) Act, 1981* along with the authorization, if required, under the *Environment (Protection) Act, 1986* while applying for ECO Mark.

### 5.5.2 Specific Requirements

**5.5.2.1** The material shall not contain any phosphate when tested as per the method prescribed in Annex D of IS 4955.

**5.5.2.2** The material shall pass the test for skin irritant and sensitization potential when evaluated as per the method prescribed in IS 11601.

**5.5.2.3** The surfactants used in the manufacture of synthetic detergent for industrial purposes shall be readily biodegradable when tested by modified Sturm test as prescribed in IS 13933.

## 6 PACKING AND MARKING

### 6.1 Packing

The material shall be suitably packed as agreed between interested parties. The packing shall be capable to prevent any moisture absorption.

**6.1.2** For ECO Mark the product shall be packed in such packages which are made from recyclable/reusable or biodegradable materials and declared by the manufacturer and may be accompanied with detailed instructions for proper use.

## 6.2 Marking

**6.2.1** The containers shall be legibly and indelibly marked with the following information:

- a) Name of the manufacturer;
- b) Name, type and recognized trade-mark of the product, if any;
- c) Net mass when packed;
- d) Batch number or lot number in code or otherwise;
- e) Month and year of manufacture;
- f) Shelf life of product;
- g) **A CAUTIONARY NOTICE:** Detergent solutions can be skin irritants. Avoid prolonged contact. Rinse garments and hands thoroughly;
- h) The following critical ingredients in descending order of quantity, percent by mass:
  - 1) Active ingredients,
  - 2) Builders,
  - 3) Soda ash,
  - 4) Fillers, and
  - 5) Enzymes, if any

In addition to above, Product may also be mark with details as per the requirement of legal metrology regulations and any other applicable statute.

### 6.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

**6.2.3** The following information shall be marked on the label for ECO Mark:

- a) List of identified critical ingredients in descending order of quantity, percent by mass [*see 6.2.1(g)*].
- b) The criteria for which the product has been labelled as ECO Mark.

NOTE — Loss in mass, even in packed condition, may occur on account of moisture loss due to environmental conditions.

## 7 SAMPLING

Representative samples of the material shall be drawn as prescribed in Annex H.

## 8 TESTS

**8.1** Tests shall be carried out as prescribed in Annexes B to G of this standard. Reference to the relevant annex of IS 4955 as applicable, are given in column 6 of Table 1.

**8.2 Quality of Reagents**

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

**Table 1 Requirements for Synthetic Detergents for Industrial Purposes**  
( *Clauses 5.4 and 8.1* )

SI No.	Characteristics	Requirements for			Method of Test Annex of IS 4955	Ref to Annex Annex of this standard
		Type 1	Type 2	Type 3		
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Active ingredient percent by mass, <i>Min</i>	35	40	20		B and C
ii)	Moisture and volatile matter content percent by mass, <i>Max</i>	5	—	—		D
iii)	Clear point, °C, <i>Max</i>	—	—	10		E
iv)	Sodium tripolyphosphate (STPP) percent by mass, <i>Max</i> ( <i>see Note 2</i> )	2.5	5	5	E	—
	Total phosphates	2.5	5	5	D	—
v)	expressed as P <sub>2</sub> O <sub>5</sub> content percent by mass, <i>Max</i> ( <i>see Notes 1 and 2</i> )					
vi)	pH of 1 percent ( <i>m/v</i> ) at 30 °C	7-9	7-9	6-8		F
vii)	Matter insoluble in water, percent by mass, <i>Max</i>	0.5	0.5	nil		G
viii)	Percent detergency, <i>Min</i>	65	55	45	G	
ix)	Ash built up, percent, <i>Max</i>	1	5	10	H	

**NOTES**

**1** Total phosphate content including phosphate based builder (as recommended in annex A) shall not exceed 2.5 percent by mass for type 1 and 5 percent by mass for type 2 and type 3.

**2** Use of Zeolite (*see* IS 15267) is recommended as an alternative and substitute to phosphate based builder.

**ANNEX A**

( Clause 5.1 and Note 1 under Table 1 )

**LIST OF CONVENTIONAL BUILDERS AND ADDITIVES**

1	Crystalline sodium aluminosilicate (zeolite)	10	Lather boosters
2	Trisodium phosphate	11	Hydrotropes
3	Sodium carbonate	12	Perfume
4	Tetra sodium pyrophosphate	13	Preservatives
5	Sodium tripolyphosphate	14	Chelating agents (sequestering agents)
6	Sodium hexametaphosphate	15	Colours
7	Sodium carboxymethyl cellulose	16	Optical brighteners
8	Sodium silicate	17	Sodium sulphate
9	Trisodium citrate	18	Enzymes

**ANNEX B**

[ Table 1, SI No. (i) ]

**DETERMINATION OF ACTIVE INGREDIENT BY CATION TITRATION****B-1 GENERAL**

In the prescribed method, the molecular mass of active matter has been taken as 342. In practice, the molecular mass of sodium alkyl benzene sulphonate varies from 337 to 347 depending on the molecular mass of alkyl benzene used for sulphonation. This method shall therefore, be used for routine analysis. In case of any dispute or doubt, the molecular mass of sodium alkyl benzene sulphonate shall be determined by the method prescribed in Annex C and which then used in calculating the active ingredient content by this method.

**B-2 OUTLINE OF THE METHOD**

A solution of the anionic detergent containing added methylene blue is shaken in chloroform, which dissolves the methylene blue salt of the detergent. The mixture is titrated with a cationic active agent, which, after it has combined with all the free anionic detergent, and begins to displace methylene blue from the salt. The end point is taken when sufficient methylene blue has been displaced into the aqueous layer to produce phases of equal colour intensity. As the reaction is not stoichiometric, it is essential to carry out standardization using a known anionic detergent similar in nature to the unknown.

NOTE — Hypochlorites and sulphites interfere with detection of the end point and should be destroyed by the addition of ferrous sulphate and hydrogen peroxide respectively.

**B-3 APPARATUS**

**B-3.1 Volumetric Flasks** — 1 000 ml, 500 ml and 250 ml capacity.

**B-3.2 Stoppered Graduated Cylinder** — 100 ml capacity.

**B-3.3 Graduated Cylinder** — 50 ml capacity.

**B-3.4 Burette** — 25 ml capacity.

**B-3.5 Pipette** — 10 ml capacity.

**B-3.6 Beaker** — 250 ml capacity.

**B-4 REAGENTS**

**B-4.1 Chloroform** — Chemically pure.

**B-4.2 Sulphuric Acid** — 5 N

Carefully add 134 ml of sulphuric acid (relative density 1.84) to 300 ml of water with constant stirring and cool to room temperature and dilute to 1 l.

CAUTION — Highly corrosive, causes severe burns use necessary safety implements (see IS 4262).

**B-4.3 Standard Sulphuric Acid** — 1.0 N

**B-4.4 Standard Sodium Hydroxide Solution** — 1.0 N

**B-4.5 Standard Sodium Lauryl Sulphate Solution** — 0.001 M



Check-up purity of sodium lauryl sulphate as given in **B-4.5.1** and simultaneously prepare the standard solution.

#### **B-4.5.1 Determination of Purity of Sodium Lauryl Sulphate**

Weigh  $5 \pm 0.2$  g of sodium lauryl sulphate to the nearest 1 mg into a 250 ml round bottom flask with ground glass neck. Add exactly 25 ml of standard sulphuric acid (*see B-4.3*) and reflux under water condenser. During the first 5 to 10 min, the solution will thicken and tend to foam strongly which is controlled by removing the source of heat and swirling the contents of the flask in order to avoid excessive foaming, instead of refluxing. The solution may be left on a boiling water bath for further 10 min till the solution clarifies and foaming ceases. Reflux for further one and half hours. Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water. Add a few drops of phenolphthalein indicator solution and titrate with standard sodium hydroxide solution. Carry out a blank test by titrating 25 ml of standard sulphuric acid (*see B-4.3*) with standard sodium hydroxide solution.

Sodium lauryl sulphate, percent by mass =

$$\frac{28.84 \times (V_1 - V_0) \times N_1}{m_1}$$

where

$V_0$  = volume of standard sodium hydroxide solution used for the blank, in ml;

$V_1$  = volume of standard sodium hydroxide solution used for the sample, in ml;

$N_1$  = normality of standard sodium hydroxide solution; and

$m_1$  = mass of sodium lauryl sulphate taken for the test, in g.

#### **B-4.5.2 Procedure**

Weigh 1.14 to 1.16 g of sodium lauryl sulphate to the nearest 1 mg and dissolve in 200 ml of water. Transfer to a ground-glass stoppered 1 l volumetric flask and dilute to the mark with water. Calculate the molarity,  $M_1$  of the solution as follows:

$$M_1 = \frac{m_2 \times p}{288.4 \times 100}$$

where

$m_2$  = mass of sodium lauryl sulphate taken, in g;

$p$  = purity, percentage by mass

#### **B-4.6 Standard Benzethonium Chloride Solution — 0.004 M**

Weigh 1.75 to 1.85 g of benzethonium chloride to the nearest 1 mg and dissolve in water. Transfer to a ground

glass-stoppered 1 l volumetric flask and dilute to the mark with water.

#### **NOTES**

**1** In order to prepare a 0.004 M solution, dry the benzethonium chloride at 105 °C. Weigh 1.792 g to the nearest 1 mg, dissolve in water and dilute to 1 l. While drying, care shall be taken that temperature should not exceed beyond 105 °C.

**2** Other cationic reagents such as cetyl trimethyl ammonium bromide and benzalkonium chloride give results identical to those obtained using benzethonium chloride. However, these tests have not been carried out in sufficient number to make it possible to state that the results will be identical no matter what the product analysed for that reason. If benzethonium chloride is not available, it is permitted to use another reagent and same should be stated in the test report. However, in case of doubt and dispute, only benzethonium chloride shall be used.

#### **B-4.7 Phenolphthalein Indicator Solution**

Dissolve 1 g of phenolphthalein in 100 ml of 95 percent (v/v) ethanol.

#### **B-4.8 Methylene Blue Solution (0.005 Percent)**

Dissolve 0.05 g of methylene blue, 50 g of sodium sulphate and 6.8 ml of concentrated sulphuric acid in water and make up the volume to 1 l with water.

#### **B-4.9 Sample Solution**

Weigh a suitable quantity of the sample in measured quantity of water so that resultant solution contains 100 to 160 mg of anionic active matter per 100 ml. About 0.65-0.70 g of sodium alkyl benzene sulphonate (based on 100 percent purity) or 4.5 g of the sample having around 16 percent anionic active matter content per 500 ml of the solution is suitable.

### **B-5 PROCEDURE**

#### **B-5.1 Standardization of Benzethonium Chloride Solution**

**B-5.1.1** Pipette 10 ml of standard sodium lauryl sulphate solution (*see B-4.5*) in a 100 ml graduated cylinder provided with a glass stopper. Add 15 ml of chloroform and 25 ml of methylene blue solution to the cylinder. Shake well. The chloroform layer (lower) will be coloured blue or greenish blue.

**B-5.1.2** From the burette, add benzethonium chloride solution slowly, initially in portions of 0.2 ml at a time. After each addition, stopper the cylinder, shake well and allow the phases to separate. Initially the chloroform phase will be coloured blue or greenish blue. Towards the end, the colour would start migrating to the aqueous layer. Note the reading at which the colour intensity in both the phases is the same when viewed under standard conditions of light, for example, against a white porcelain tile, under normal day light.

**B-5.1.3** Calculate the molarity of benzethonium chloride solution as follows:

Molarity of benzethonium chloride solution,

$$M_2 = \frac{10 M_1}{V_1}$$

where

$M_1$  = molarity of sodium lauryl sulphate solution (B-4.5.2); and

$V_1$  = volume of benzethonium chloride solution added, in ml.

#### B-5.2 Determination of Anionic Active Matter

Take 10 ml of the sample solution (B-4.9) instead of sodium lauryl sulphate solution and proceed as described in B-5.1.

**B-5.3** Calculate the anionic active matter as sodium alkyl benzene sulphonate as follows:

Anionic active matter, percent by mass =

$$\frac{342 \times V_2 \times M_2 \times 5}{m_2}$$

where

342 = molecular mass of sodium alkyl benzene sulphonate taken for calculation;

$V_2$  = volume of benzethonium chloride solution added, in ml;

$M_2$  = molarity of benzethonium chloride solution (B-5.1.3); and

$m_2$  = mass of the sample, in g.

## ANNEX C

[ Table 1, SI No. (i) and Clause B-1 ]

### DETERMINATION OF MOLECULAR MASS OF SULPHONIC ACID OR SODIUM SALT OF SULPHONIC ACID

#### C-1 APPARATUS

**C-1.1 Beakers** — 150 and 1 000 ml capacity

**C-1.2 Buchner Flask** — 500 ml capacity, fitted with a sintered glass filter funnel (porosity 4).

**C-1.3 Separating Funnels** — 1 000 ml capacity

**C-1.4 Wide-Mouthed Flat-Bottom Flask** — 200 ml capacity

**C-1.5 Air-Oven** — preferably electrically heated with temperature control and display device

**C-1.6 Steam Bath**

#### C-2 REAGENTS

**C-2.1 Caustic Soda Solution** — 10 percent ( $m/v$ )

**C-2.2 Ethyl Alcohol** — 30 percent, 96 percent and absolute ( $v/v$ )

**C-2.3 Diethyl Ether**

**C-2.4 Acetone**

**C-2.5 Phenolphthalein Indicator** — 1 percent solution in 95 percent ( $m/v$ ) ethyl alcohol

**C-2.6 Methyl Orange Indicator** — 0.1 percent ( $m/v$ )

**C-2.7 Ferric Ammonium Sulphate Indicator** — Saturated solution

**C-2.8 Standard Sulphuric Acid** — Approximately 0.1 N

**C-2.9 Standard Silver Nitrate Solution** — Approximately 0.1 N

**C-2.10 Standard Ammonium Thiocyanate Solution** — Approximately 0.1 N

**C-2.11 Nitric Acid** — Concentrated, relative density 1.42

**C-2.12 Nitrobenzene**

#### C-3 PROCEDURE

**C-3.1** Weigh about 3 g of the material into a 150 ml beaker. Dissolve in minimum quantity of water and neutralize with caustic soda solution, if required. Evaporate on a steam bath to almost complete dryness. Digest with 50 ml of 96 percent ethyl alcohol by heating on a steam bath for about 2 min. Stir and break up any hard lumps with a glass rod flattened at one end. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30 ml portions of boiling ethyl alcohol. Filter each extract in turn through the same sintered glass funnel and finally, wash the residue five times with hot ethyl alcohol to remove all the alcohol soluble. Evaporate the combined filtrate approximately 50 ml in an evaporating dish and transfer it to a



separating funnel. Rinse the evaporating dish once with 50 ml of 96 percent ethyl alcohol and then four times with 50 ml portions of water. Add each wash in turn to the separating funnel. Add 150 ml of diethyl ether, swirl gently to ensure adequate mixing, and allow the two phases to separate. Run off the aqueous alcoholic layer into a second separating funnel, and extract twice with 75 ml portions of diethyl ether. Transfer the aqueous alcoholic phase into a beaker, and combine the three ether extracts.

**C-3.2** Take the combined ether extracts in a clean separating funnel. Wash three times successively with 50 ml portions of 30 percent ethyl alcohol and then successively with 50 ml portions of water until the ether phase is free from alcohol, usually 7 to 10 water washes are necessary. Combine all the alcoholic and aqueous extracts, neutralize to phenolphthalein and evaporate on a steam bath until the volume is reduced to about 25 ml. Add an equal volume of absolute alcohol and evaporate to dryness. The solution should remain pink to phenolphthalein throughout evaporation. To ensure that the residue is completely anhydrous, add 30 ml of hot absolute alcohol and again evaporate to dryness. Extract the residue with 30 ml of hot 96 percent ethyl alcohol, stirring and breaking up the solid matter in the dish with a glass rod. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Extract the residue in the dish with further six consecutive 30 ml portions of hot 96 percent alcohol. Pass each extract in turn through the sintered glass filter. Finally, wash the residue in the sintered glass filter three times with about 20 ml of hot 96 percent ethyl alcohol from the jet of a wash bottle.

**C-3.3** Transfer the filtrate and washing in the Buchner flask to a wide-mouthed flat-bottom flask, evaporate nearly to dryness on a water-bath, and drive off the remaining solvent by directing a gentle stream of dry air into the flask whilst continuously rotating the latter on the water bath, a thin film of active matter, easy to dry, is thereby obtained. Add 10 ml of acetone, evaporate and remove the last traces of solvent as prescribed above, cool in a desiccator and weigh. Heat the flask for not more than 5 min in an air oven at a temperature of  $100 \pm 1^\circ\text{C}$ , gently blow out with a current of air, cool and re-weigh. Repeat this drying process until the difference between two successive weighing does not exceed 3 mg.

**C-3.4** The extract obtained contains active matter, some sodium chloride and possible traces of alkali carbonates which may have passed through the filter in the presence of the detergent. Find the percentage of sodium carbonate and sodium chloride in the extract by

using a portion of the extract as prescribed in **C-3.4.1** and **C-3.4.2** respectively.

#### **C-3.4.1** *Determination of Alkali Carbonates*

Weigh accurately about 1 g of the extract. Dissolve it in cold water, add a few drops of methyl orange indicator and titrate with standard sulphuric acid to methyl orange end point.

##### **C-3.4.1.1** *Calculation*

Mass of sodium carbonate, in g =

$$\frac{0.053 \times V_1 \times N_1 \times m_1}{m_2}$$

where

$V_1$  = volume of standard sulphuric acid solution used, in ml;

$N_1$  = normality of the standard sulphuric acid solution;

$m_1$  = mass of the total extract, in g; and

$m_2$  = mass of the extract taken for analysis, in g.

**C-3.4.1.2** Reserve the solution for the estimation of chlorides.

#### **C-3.4.2** *Determination of Chlorides*

**C-3.4.2.1** To the solution remaining after the estimation of alkali carbonate (*see C-3.4.1.2*) add 2 ml of concentrated nitric acid and 20 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene and shake vigorously. Titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate as indicator.

##### **C-3.4.2.2** *Calculation*

Mass of sodium chloride, in g =

$$\frac{0.0585 \times (20N_3 - V_2N_2) \times m_1}{m_2}$$

where

$N_3$  = normality of standard silver nitrate solution;

$V_2$  = volume of standard ammonium thiocyanate solution used, in ml;

$N_2$  = normality of standard ammonium thiocyanate solution;

$m_1$  = mass of the total extract (*see C-3.3*), in g; and

$m_2$  = mass of the extract taken for analysis in **C-3.4.1.1**.

**C-3.5** Weigh accurately about 0.65-0.70 g of the extract. Dissolve in water and make up to 500 ml. Follow the titration procedure given in **B-5.1** taking 10 ml of the solution for titration.

**C-4 CALCULATION**

Molecular mass of sodium salt of sulphonic acid =

$$\frac{(100 - M_1 - M_2) \times M}{5 \times V_1 \times T_1}$$

where

$M_1$  = percent by mass of sodium carbonate

(see C-3.4.1);

$M_2$  = percent by mass of sodium chloride  
(see C-3.4.2);

$M$  = mass of the extract taken, in g;

$V_1$  = volume of benzethonium chloride solution added, in ml; and

$T_1$  = molarity of benzethonium chloride solution.

**ANNEX D**

[ Table 1, SI No. (ii) ]

**DETERMINATION OF MOISTURE AND VOLATILE MATTER****D-1 APPARATUS**

**D-1.1 Porcelain or Silica Dish** — 6 to 8 cm in diameter and 2 to 4 cm in depth.

**D-1.2 Desiccator** — Containing an efficient desiccant, such as phosphorus pentoxide.

**D-1.3 Air-Oven** — Preferably electrically heated, with temperature control and display device capable to maintain temperature  $105 \pm 1$  °C.

**D-2 PROCEDURE**

Weigh accurately about 5 g of the material into a dry tared dish and dry to constant mass in an air-oven at

a temperature of  $105 \pm 1$  °C. Cool in a desiccator and weigh. Constant mass shall be considered to have been attained when successive heating for one-hour period shows a difference of not more than 5 mg in the net loss in mass.

**D-3 CALCULATION**

Moisture and volatile matter content, percent by mass =

$$\frac{(M - m) \times 100}{M}$$

where

$M$  = mass of the material taken for the test, in g; and

$m$  = mass of the material after drying, in g.

**ANNEX E**

[ Table 1, SI No. (iii) ]

**DETERMINATION OF CLEAR POINT TEMPERATURE****E-1 GENERAL**

**E-1.1** This method is not applicable to materials showing reverse solubility temperature effect.

**E-1.2** The clear point is defined as the temperature at which clearing occurs under the conditions of the test.

**E-2 APPARATUS**

**E-2.1 Beaker** — 2 l capacity to serve as a water bath.

**E-2.2 Wide-mouthed Bottle** — 450-ml capacity, height 190 mm and inside diameter of neck 38 mm.

**E-2.3 Test Tube** — 100 mm in length and 25 mm in diameter.

**E-2.4 Stirrer** — Made of stainless steel or glass with one end bent in the form of a loop of 19 mm outside diameter.

**E-2.5 Thermometer** — Range  $-10$  °C to  $100$  °C, least count  $1$  °C.

**E-2.6 Cooling Mixture** — Isopropyl alcohol or ethanol to which solid carbon dioxide or ice-salt mixture is added as required.

**E-3 PROCEDURE**

**E-3.1** Place 25 ml of the liquid product itself in one of the test tubes and cool it whilst carefully stirring to avoid incorporation of air bubbles, until solid material

separates, and the material under test becomes solid and opaque.

**E-3.2** If no solid material separates after cooling up to 0 °C, the test shall be discontinued and the sample shall be taken as passing the requirement of the test.

**E-3.3** If solid material separates during cooling up to 0 °C, heat the bath gradually whilst stirring, until the solution becomes clear. Note the temperature and record it as the clear point of the solution.

## ANNEX F

[ Table 1, SI No. (vi) ]

### DETERMINATION OF pH

#### F-1 GENERAL

pH determination shall be made in an acid-free atmosphere.

#### F-2 APPARATUS

##### F-2.1 pH Meter

Any standard electrometric instrument, equipped with a low sodium error glass electrode. The instrument shall be calibrated and standardized with standard buffer solution (see F-3.2) before use.

**F-2.2 Volumetric Flask** — 1 000 ml capacity.

**F-2.3 Beaker** — 100 ml capacity.

#### F-3 REAGENTS

##### F-3.1 Distilled Water

Distilled water shall be boiled thoroughly or purged with carbon dioxide-free air to remove carbon dioxide, and shall be protected with soda lime or soda carbon

dioxide. It shall be protected with soda lime or soda asbestos while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 30 °C. There shall be no residue on evaporation when heated at 105 °C for 1 h shall not exceed 0.5 mg/1.

##### F-3.2 Standard Buffer Solution

Any two suitable buffer solutions within the pH range of 9 to 11 at 30 °C shall be used for calibration of pH meter.

#### F-4 PROCEDURE

Weigh  $10 \pm 0.001$  g of the material and transfer to all volume tric flask. Partially fill the flask with distilled water and agitate until the sample is completely dissolved. Adjust the temperature of the solution and the distilled water to  $30 \pm 0.5$  °C, and fill up to the mark with distilled water. Stopper the flask, mix thoroughly, and allow the solution to stand at a temperature of 30 °C for two hours prior to measuring the pH. Measure the pH of the solution using a glass electrode.

## ANNEX G

[ Table 1, SI No. (vii) ]

### DETERMINATION OF MATTER INSOLUBLE IN WATER

#### G-1 PROCEDURE

**G-1.1** Weigh accurately about 5 g of the material into a beaker and digest with 50 ml of ethyl alcohol by heating on a steam-bath for about 2 min. Stir and break up any hard lump with a glass rod flattened at one end. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel no. 4 fitted to a Buchner flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30 ml portions of

boiling ethyl alcohol. Filter each extract in turn through the same sintered glass funnel and, finally, wash the residue several times with hot ethyl alcohol to remove all the alcohol solubles.

**G-1.2** Change the receiver, extract the residue with successive portions of distilled water at about 60 °C, and wash the residue several times to remove all the water solubles. Dry the sintered glass funnel with the residue in an air-oven at a temperature of  $105 \pm 2$  °C until constant mass is obtained.

**G-1.3 Calculation**

Matter insoluble in water, percent by mass =  $\frac{m}{M} \times 100$

where

$m$  = mass of the matter insoluble in water, in g; and

$M$  = mass of the material taken for the test, in g.

**ANNEX H**

( Clause 7 )

**SAMPLING PROCEDURE FOR SYNTHETIC DETERGENTS****H-1 GENERAL REQUIREMENTS**

**H-1.1** Following precautions shall be taken in drawing, preparing, storing and handling the samples:

**H-1.1.1** Samples shall be taken in a protected place which should not be exposed to atmospheric air.

**H-1.1.2** The sampling instruments shall be clean and dry.

**H-1.1.3** The samples, material being sampled, the sampling instruments and the containers for such samples shall be protected from adventitious contamination.

**H-1.1.4** The sample shall be placed in clean and dry glass containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

**H-1.1.5** Each container shall be sealed air-tight after filling, and marked with full details of sampling, date of sampling batch or code number, name of manufacturer, and other important particulars of the consignment.

**H-1.1.6** The samples shall be stored in such a manner that the temperature of the material does not vary markedly from ambient temperature. Samples should be protected from light.

**H-2 SCALE OF SAMPLING****H-2.1 Lot**

In a single consignment, all the packages containing material of the same grade and drawn from the same batch of manufacturer, shall constitute a lot. If the consignment consists of packages containing material of different grades or batch of manufacture, then the packages containing detergent of the same grade and batch of the manufacture shall be grouped together and each such group shall constitute a separate lot.

**H-2.2** For ascertaining the conformity of the lot to the requirements prescribed in this standard, tests shall be carried out on each lot separately. The number ( $n$ ) of packages to be selected for drawing samples shall

depend upon the size ( $N$ ) of the lot and shall be in accordance with Table 2.

**H-2.3** The packages shall be selected at random. In order to ensure the randomness of selection, a random number table shall be used. For guidance and use of random number tables, IS 4905 may be used. In the absence of a random number table, the following procedure may be adopted:

‘Starting from any package in the lot, count them in one order as 1, 2, 3 up to  $r$  and so on where  $r$  is the integral part of  $N/n$  ( $N$  being the lot size and  $n$  the number of packages to be selected). Every  $r$ th package thus counted shall be withdrawn to give the required sample size.

**Table 2 Scale of Sampling**

( Clause H-2.2 )

No. of Packages in the Lot ( $N$ )	No. of Packages to be Selected ( $n$ )
4 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

NOTE — When the size of the lot is 3 packages or less, the number of containers to be selected and the criteria for judging the conformity of the lot to the specification shall be as agreed between the interested parties.

**H-3 PREPARATION OF GROSS SAMPLES, TEST SAMPLES AND REFEREE SAMPLES****H-3.1 Gross Samples**

**H-3.1.1.1** From each one of the packages as selected in **H-2** draw one or more containers randomly. The material in the containers so chosen shall be nearly thrice the quantity required for the test as indicated in **H-4**.

**H-3.1.1.2** The material from the containers as selected in **H-3.1.1.1** shall be mixed thoroughly to give the gross sample for the package.

### H-3.2 Test Samples

**H-3.2.1** Segregate carefully the gross samples (see **H-3.1.1.1**) of powders. From the gross sample, take a small but equal quantity of material and mix it thoroughly into a composite sample which should be of a size sufficient to carry out triplicate testing for all the characteristics specified under **H-4**. The composite sample shall be divided into three equal parts; one for the purchaser, another for the supplier, and the third for the referee.

**H-3.2.2** The remaining portion of the material in each one of gross samples shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the 'n' selected packages shall be for the purchaser, another for the supplier, and the third for the referee.

**H-3.2.3** All the composite and individual samples shall be transferred to separate containers. These containers shall be air-tightly sealed with stoppers, and labelled with full particulars of identification given in **H-1.1.5**.

### H-3.3 Referee Samples

**H-3.3.1** The referee samples shall consist of a composite sample and a set of 'n' individual samples. All the containers shall bear the seals of interested parties and shall be kept at a place agreed to between the two parties.

**H-3.3.2** Referee samples shall be used in case of any dispute between the purchaser and the supplier.

### H-4 NUMBER OF TESTS

**H-4.1** Tests for the determination of characteristics of active ingredient shall be performed on each of the individual samples.

**H-4.2** Tests for determination of the remaining characteristics specified in Table 1 shall be conducted on the composite sample.

### H-5 CRITERIA FOR CONFORMITY

#### H-5.1 For Individual Samples

For the characteristics which have been determined on the individual samples, the mean (X) and the range (R) of the test results shall be calculated as follows:

$$\text{Mean (X)} = \frac{\text{Sum of test results}}{\text{Number of test results}}$$

Range (R) = the difference between the maximum and the minimum value of the test results.

- a) The value of the expression  $(X - K R)$  shall be calculated from the relevant test results [see also **H-5.1 (b)**]. If the value so obtained is greater than or equal to minimum limit, the lot shall be declared as conforming to the requirement for that characteristic.
- b) The value of the factor K referred to in **H-5.1 (a)** shall be chosen in accordance with Table 3, depending upon the acceptable quality level that is the percentage of nonconforming packages that may be tolerated reasonably.

**Table 3 Value of 'K' for Achieving Different Quality Levels**

[ Clause H-5.1 (b) ]

Acceptable Quality Level	Value of 'K'
(1)	(2)
Not more than 3.0 percent defectives	0.4
Not more than 1.5 percent defectives	0.5
Not more than 0.5 percent defectives	0.6

#### H-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all the remaining characteristics determined on composite sample, the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1 of this standard.

## ANNEX J

( Foreword )

### COMMITTEE COMPOSITION

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

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